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Effect of Nanofiller Characteristics on Nanocomposite Properties

*Dennis C. Working, Peter T. Lillehei, Sharon E. Lowther, and Emilie J. Siochi
Langley Research Center, Hampton, Virginia*

*Jae-Woo Kim, Godfrey Sauti, Kristopher E. Wise and Cheol Park
National Institute of Aerospace, Hampton, Virginia*

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National Aeronautics and
Space Administration

Langley Research Center
Hampton, Virginia 23681-2199

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INTRODUCTION

Carbon nanotubes (CNT) are essentially sp^2 bonded graphene sheets of carbon rolled into cylinders having large aspect ratios, yielding nanotubes possessing novel mechanical, electrical and thermal properties. The number of concentric cylinders in each nanotube determines their designation as single wall carbon nanotubes (SWCNT), double wall (DWCNT), few wall (FWCNT) or multiwall (MWCNT). While SWCNTs are the most desirable of these classes of CNTs, they are also the most difficult and therefore expensive to produce in pure form.

The exponential rise in reported work for these high aspect ratio hollow carbon nanotubes was sparked by Iijima's report on multiwall carbon nanotubes in 1991 [1]. In recent years, CNT related publications have reached over 9,000/year, translating to about 25 papers a day on the subject of carbon nanotubes alone. A considerable number of these publications report endeavors to gain a fundamental understanding of carbon nanotubes, their synthesis, characterization and properties. Papers on the applications of CNTs are significantly fewer. For instance, about 2000 of those publications are in the area of electronics, which is advancing most rapidly due to the drive for smaller and smaller electronic devices in the marketplace. The area of nanocomposite development, a subject with direct implications on the requirement for weight reduction in aerospace structures, has publications numbering about 1000 in the last couple of years, with reports related to mechanical and electrical properties for these materials comprising a few hundred per year.

Much of the interest in CNTs is spurred by the anticipation that their incredible suite of properties will enable applications having sweeping societal impact; however, an understanding of production conditions necessary to make large-scale homogeneous batches of material consistently remains elusive [2]. Batch to batch inconsistencies limit the applications of these materials in aerospace structures where certification requirements are rather stringent. However, while efforts to consistently produce pure CNTs persist, the potential applications of carbon

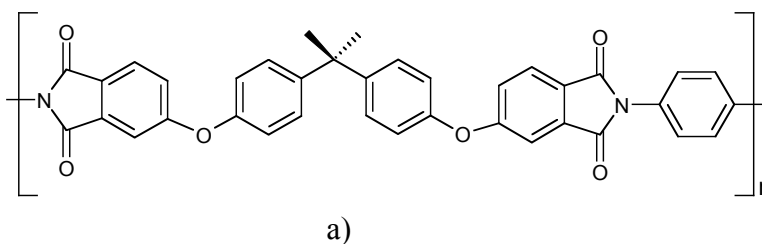
nanotube nanocomposites are already being assessed. The first report on fabrication of nanocomposites from CNTs came out only three years after Iijima's paper [3].

For aerospace applications, the combination of density, mechanical, electrical and thermal properties of CNTs opens up the possibility of designing efficient, lightweight, multifunctional structures as a route to weight reduction in structures. Inconsistencies in CNT quality have hampered advances in this area, as CNT characteristics influence their dispersion, alignment and matrix/CNT interface quality and have significant impact on nanocomposite performance [4,5,6]. Some of these factors will be examined in the survey of nanofillers presented here. The objective of this work is to explore the influence of dimensionality, size and filler chemistry on the physical properties of nanocomposites. Two different high-performance polymer matrices are used. Results shown here are preliminary and reflect the interim nature of this report.

EXPERIMENTAL

Matrix

Two polyimide compositions were used as polymer matrices for this study. Ultem[®] 1000 was obtained from GE Plastics in pellet form and LaRC[™]-8515 powder synthesized at a stoichiometric offset of 5% was purchased from Imitec, Inc. Both materials were dried under vacuum at 150°C and stored in a dessicator prior to use. Their chemical structures are shown in Figure 1.



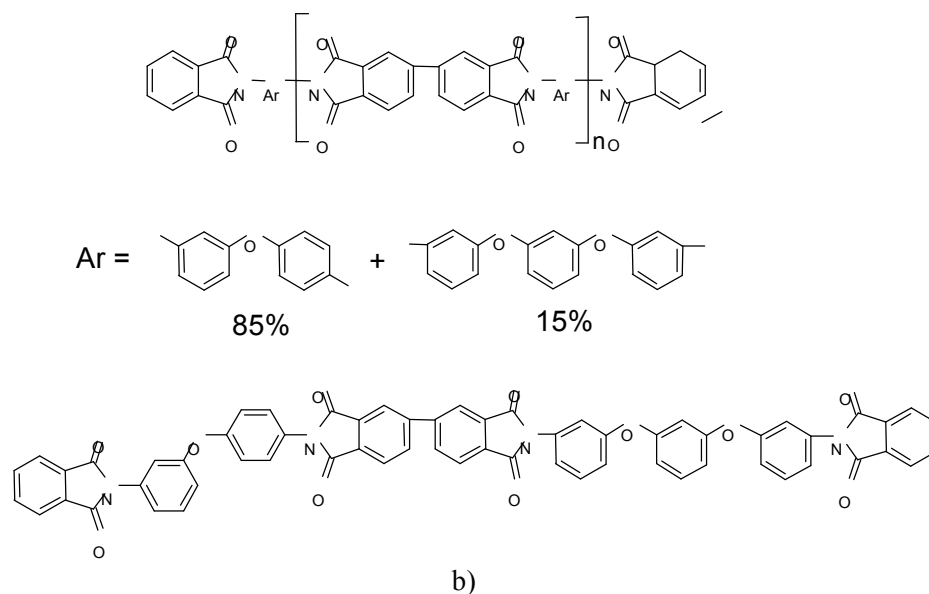


Figure 1: Molecular structures of a) Ultem[®] and b) LaRC 8515.

Nanofillers

Ten fillers having a range of dimensions and sizes were used. The sources and pretreatment of these materials are shown in Table 1.

Processing

Forty compositions of nanocomposites were fabricated for this study. Nanofillers were mixed with each matrix material at 0.3 and 1.0 wt%. The components of the processing equipment used included a 7.5 hp C.W. Brabender PL2000 with 6 zone control, an electrically heated, air cooled 3 zone 45/60 melt mixer equipped with roller blades and a half size electrically heated single zone mixer.

Melt Mixing

Approximately 50 grams of matrix material and the appropriate amount of nanofiller were weighed in a pan and poured into the melt mixer, which has been thermally equilibrated at 300°C under a nitrogen blanket. The mixer was then brought up to 25 rpm under a nitrogen purge of approximately 2 cc/min at 1.38×10^5 Pa (20 psi). Mixing was performed for 3 hours before the material was removed for further processing.

TABLE 1
Nanofiller Sources and Pretreatment

Nanofiller	Source	Pretreatment
Carbo Spheres	Firefox Enterprises, Inc.	Sifted with 120 μ m mesh
Silica microspheres	Eccospheres, STM Y311-2	Sorted and stored at 150°C prior to use
HiPco Single Wall Carbon Nanotubes, Purified	Carbon Nanotechnologies Inc.	Used as received
Free Electron Laser Single Wall Carbon Nanotubes	Jefferson Laboratory	Used as received
Few Wall Carbon Nanotubes, Purified	Duke University	Used as received
Multiwall Carbon Nanotubes	Duke University	Used as received
Carbon Nanofibers	Pyrograf III Carbon Fiber, HT Grade, from Applied Sciences Inc.	Used as received
Silica Fibers	Thermal Products Company, Inc., SILTEMP 84S	Baked at 538°C overnight.
Thermally Exfoliated Graphite Oxide	Lot # 5-b from Princeton University, BIMAT URETI	Used as received
Functionalized Graphene Sheets	Lot 17-C from Princeton University, BIMAT URETI	Used as received

Degassing

Melt mixed samples were transferred to stainless steel pans treated with a very thin film of release agent to prevent sticking. The pans were placed in a vacuum oven that was brought up to 240°C for Ultem[®] blends and 250°C for LaRC 8515 under vacuum. Samples were degassed for ~24 hours, before the temperature was reduced to less than 100°C. The oven was back filled with nitrogen to ambient pressure, and samples were removed, then bagged and stored in dry boxes.

Molding

Melt mixed nanocomposite samples were ground up using a Dynisco Polymer Test grinder fitted with a 5 mm mesh screen, just before molding, to minimize exposure of large surface areas to air. Molding of dogbone specimens was done in a 4 cc capacity Dynisco Polymer Test Laboratory Mixing Molder Model LMM-4-120. Watershield[™] was used as the release agent on the stainless steel pans and molds. The ground pellets were placed in the sample bowl set at 250°C and tamped down to reduce entrapped gas in the bowl. The bowl temperature was then brought to 135°C above glass transition temperature (T_g) for each matrix – 350°C for Ultem[®] and

360°C for LaRC™-8515, respectively. Mold temperatures were 300°C for Ultem® and 310°C for LaRC™-8515, or 85°C above T_g . After each composition was processed, the bowl was purged and the ram scraped clean, while the parts were still at temperature. The bowl temperature was then dropped to the tamping temperature to fill with next sample. The bowl and molds were baked clean between processing of the two matrix resins.

CHARACTERIZATION

Density Measurements

Densities were measured using the density accessory that came with the Mettler Toledo AG285 analytical balance. Calculations were based on the displacement method. All samples were measured within a few hours of molding. The densities were used to calculate specific moduli.

Mechanical Testing

Mechanical properties of the dogbone specimens prepared as described above were measured on an Instron model 5848 Microtester. Tensile properties were determined following ASTM D882 (Standard Test Method for Tensile Properties of Thin Plastic Sheeting). The gage length of the specimens was 10 mm and the strain rate used was 5 mm/min with a 1 kN load cell.

Electrical Conductivity

Samples were cut into blocks having a rectangular cross-section of approximately 15mm x 13mm (actual dimensions of each sample were measured to calculate the geometric factor). The sample surface was cleaned with isopropanol. An electron beam evaporator was used to deposit gold electrodes onto the sample surfaces in the following sequence: 100 Å of Chromium (to aid in electrode binding) and then 300-350 Å of gold. All electrical measurements were carried out at room temperature on a Novocontrol Broadband Dielectric Converter + Solartron SI1260 system. Data acquisition was performed with Novocontrol WinData software.

Microscopy

All nanomaterials were examined under a Hitachi S-5200 high-resolution scanning electron microscope (HR-SEM) using either the secondary electron (SE) detector or the transmission electron (TE) detector. Imaging the nanofillers in transmission mode via TE is akin to an examination using a transmission electron microscope (TEM).

The nanocomposite samples were examined both on the surface and in cross-section in the HR-SEM to determine the nature of the dispersion of the nanofiller throughout the polymer matrix. The interfacial regions around the nanofillers were closely examined to determine the degree to which the polymer wetted the nanofiller surface. These micrographs will help elucidate aggregation, wetting and dispersion occurring at the nanofiller/matrix interface.

RESULTS

Nanofillers

A summary of the characteristics of the nanofillers is shown in Table 2. Properties that are hypothesized to have significant effects on the mechanical, electrical and thermal properties of interest include the filler dimension, chemistry and size. The nanofillers were classified into three categories depending upon their dimensional order. Zero dimensional spherical particles were classified as 0D, one dimensional tubes and rods as 1D and two dimensional sheets as 2D.

Carbospheres and silica microspheres were classified as 0D. Carbospheres are low density (0.198 g/cc), conductive carbon hollow spheres capable of withstanding temperatures well in excess of 4000 °F, while silica microspheres are insulating. The 0D spheres varied widely in diameter from 10 to 120 μm . Before processing, they were sifted using a 120 μm mesh to remove larger particles or agglomerates.

The 1D fillers used were carbon and silica-based materials including SWCNT, FWCNT, MWCNT, carbon nanofibers (CNF), and silica fibers (SF). The HiPCO SWCNTs are small diameter nanotubes (~ 1 nm) that have been highly purified and have an iron catalyst content of approximately 10 wt%. Unpurified SWCNT fabricated using the free electron laser ablation

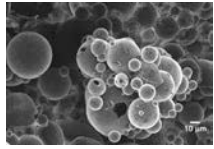
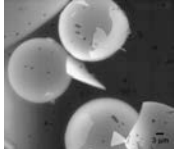
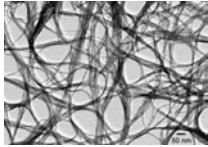
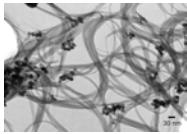
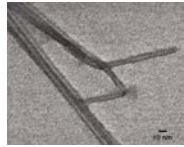
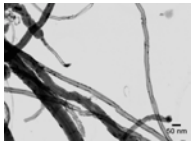
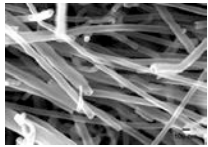
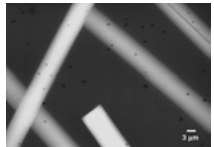
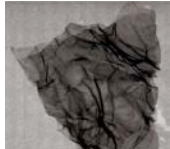
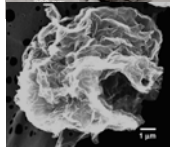
method (FEL) had diameters ranging from 1 to 1.4 nm and typical lengths in the range of 5 to 20 microns. As received, the nickel-cobalt (NiCo) catalyst content was approximately 1 to 4 atomic%. Purified FWCNT provided by Duke University consisted of nanotubes having an average diameter of 2 nm, while the MWCNT used had diameters ranging from ~ 10 to 20 nm. While the diameters of the 1D materials varied from 1 nm to 100 nm for the carbon-based fillers, the silica fibers had a uniform diameter of 10 μm . Many groups use CNF as a low-cost substitute for nanotubes. They are fabricated by the graphitization of chemically vapor deposited carbon and thus contain a negligible quantity of iron catalyst. They have diameters ranging from 70 to 200 nm and lengths estimated between 30 to 100 microns. The range of physical characteristics possessed by the set of 1D nanofillers used should permit a comparison of the influence of physical properties on the mechanical, electrical and thermal properties of the nanocomposites produced therefrom.

The fillers classified as 2D were a thermally exfoliated graphite oxide (TEGO) and functionalized graphene sheets (FGS). TEGO had smaller sheet sizes ranging from 500 nm to 2 μm and a thickness of about 2 nm, while the FGS was larger, with sizes ranging from 5 to 20 μm with a 20 nm thickness.

Mechanical Properties of Nanocomposites

The tensile moduli measured for the four sets of nanocomposites (Ultem[®] and LaRC[™]-8515 doped with 0.3 wt% and 1.0 wt% of all the nanofillers listed in Table 1) were normalized using the density to obtain specific moduli. The comparison of specific moduli is summarized in Figures 2 to 5. The data are plotted as % increase in specific moduli for the doped materials relative to the neat matrix resin. Figure 2 shows the effect of doping concentration on the specific modulus of Ultem[®]. Except for FWNT and FGS, the general trend is a greater enhancement of specific modulus for the 0.3% doped nanocomposite compared to the 1.0% nanocomposite. The analogous plot for LaRC[™]-8515 nanocomposite is shown in Figure 3. The results for LaRC[™]-8515 are dependent on the dimension of the fillers. The nanocomposites doped with the

TABLE 2
Filler Characteristics

Filler	Filler Dimension	Composition	Size	Morphology
Carbospheres	0 D	Graphitic	10 ~ 100 μm	
Silica Microspheres	0 D	SiO_2	15 ~ 120 μm	
SWNT (HiPCO)	1D	Carbon	~ 1 nm	
SWNT (FEL)	1D	Carbon	< 2 nm	
FWNT	1D	Carbon	2 ~ 10 nm	
MWNT	1D	Carbon	10 ~ 20 nm	
Carbon Nanofibers	1D	Carbon	~ 100 nm	
Silica Fibers	1D	SiO_2	10 μm	
Thermally Exfoliated Graphite Oxide (TEGO)	2D	Graphitic Oxide	500 nm ~ 2 μm / ~ 2 nm	
Functionalized Graphene Sheets (FGS)	2D	Graphitic Oxide	5 ~ 20 μm / ~ 20 nm	

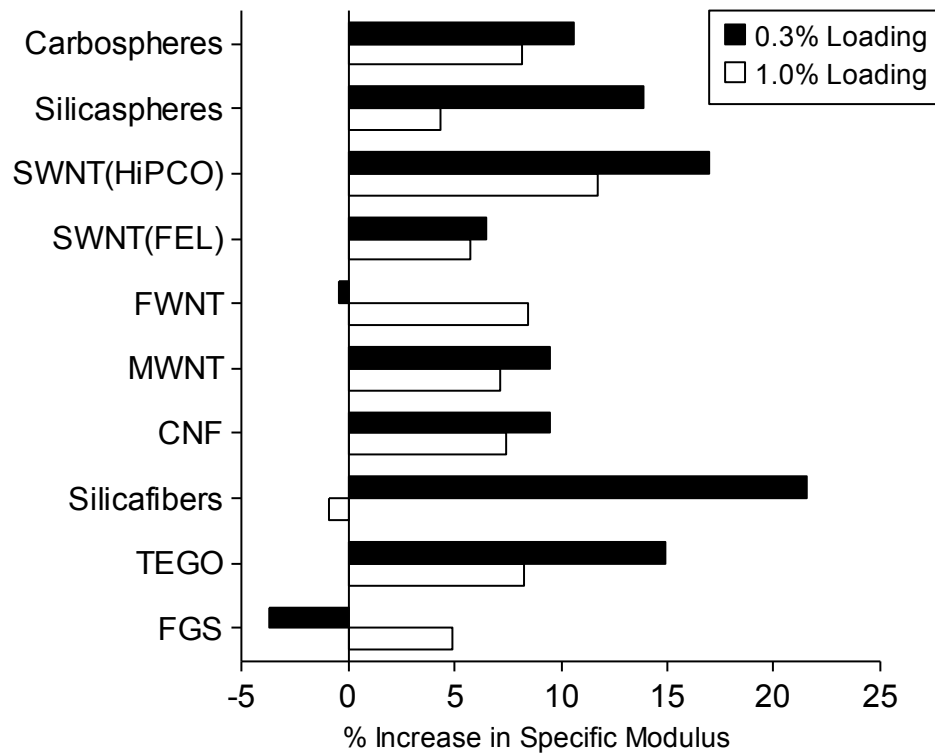


Figure 2: Effect of filler concentration on specific modulus of Ultem[®] nanocomposite.

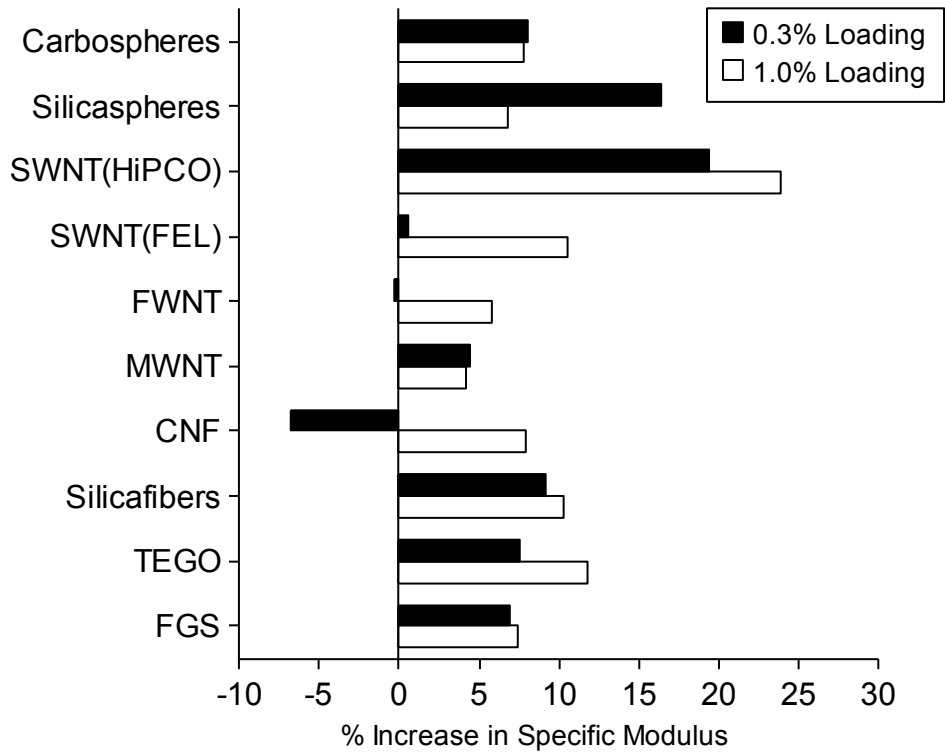


Figure 3: Effect of filler concentration on specific modulus of LaRC[™]-8515 nanocomposite.

spherical fillers have slightly lower moduli at the higher doping level. In comparison, the dominant trend followed by all other materials is a greater enhancement of moduli as the filler concentration increased.

The expected trend for these materials is an increase in specific modulus with increasing filler concentration. The results for Ultem[®] may be anomalous. A neat resin batch was processed with each set of nanocomposites. The modulus measured for the neat Ultem[®] resin processed at the same time as the 0.3% nanocomposites was much lower than the one measured for the resin processed with the 1.0% nanocomposite set. This resulted in an exaggerated reinforcement effect manifested in the specific moduli and suggests that unoptimized processing conditions may have influenced the results. In an effort to reduce the number of independent variables in the fabrication of these samples, processing conditions were chosen so that there was some uniformity in the parameters used for Ultem[®] and LaRC[™]-8515. Unfortunately, this resulted in

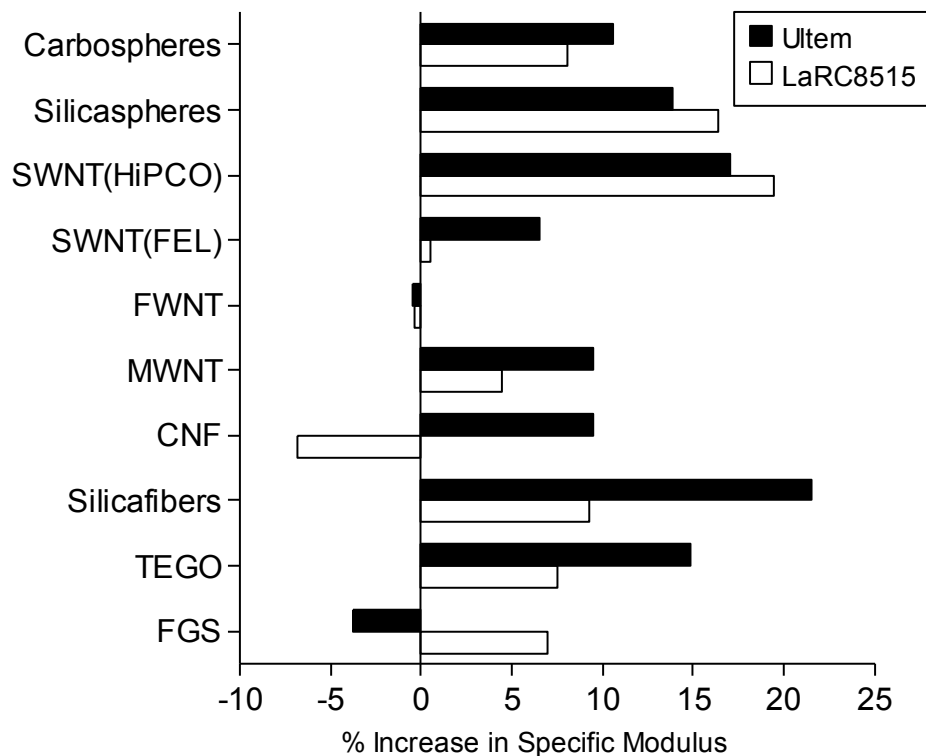


Figure 4: Effect of matrix chemistry on specific modulus of 0.3% nanocomposites.

conditions not being optimum for either system. Perhaps the results would be different if the conditions used were optimized for each matrix instead. This will be considered for future work.

The problem with the 0.3wt% Ultem[®] data, described in the previous paragraph, is evident again in Figure 4. This plot shows a much larger improvement in modulus for the Ultem[®] matrix composites than for the LaRC[™]-8515 composites. Just the opposite is seen in Figure 5, which shows that, at 1.0wt% loading, all fillers improved the specific modulus of the LaRC[™]-8515 matrix more than they do for Ultem[®].

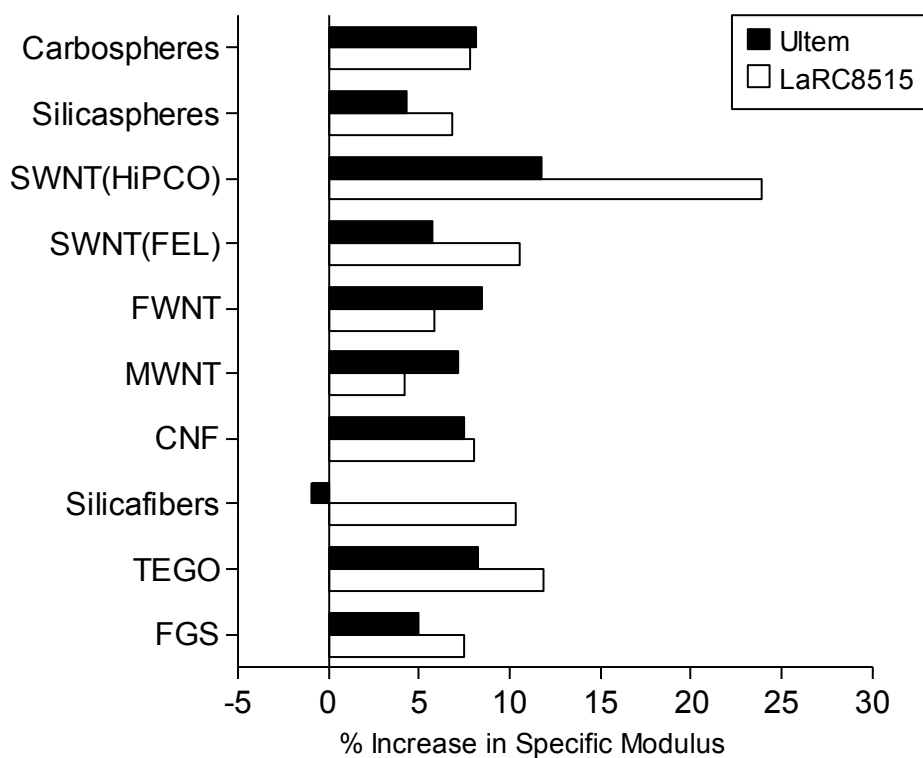


Figure 5: Effect of matrix chemistry on specific moduli of 1% nanocomposites.

A comparison of specific moduli results for the 1% nanocomposites shows that in most cases, the LaRC[™]-8515 nanocomposite moduli were enhanced more by the nanofillers compared to the Ultem[®] nanocomposites. An examination of the filler/matrix interface as shown in Figure 6 suggests that the matrix chemistry has an impact on the quality of this interface, thus influencing the reinforcement observed. Images shown here compare the dispersion of HiPco SWCNT, FWNT, MWNT and CNF in Ultem[®] and LaRC[™]8515. Note that in every case, the nanofillers

dispersed better in the LaRC™-8515 matrix compared to the Ultem® matrix. Qualitatively, this is demonstrated by less aggregation of the same concentration of nanofiller in the LaRC™-8515 matrix compared to the Ultem® matrix. Due to greater aggregation in the Ultem® nanocomposite, these materials had a larger proportion of resin rich areas compared to the LaRC-8515 nanocomposites. The interfaces between the matrix and the fillers were also voidier in the Ultem® nanocomposites and wetting of the filler was poorer, resulting in less intimate contact between the filler and the matrix. This is most pronounced in the case of the Carbosphere filled samples as shown in Figure 7.

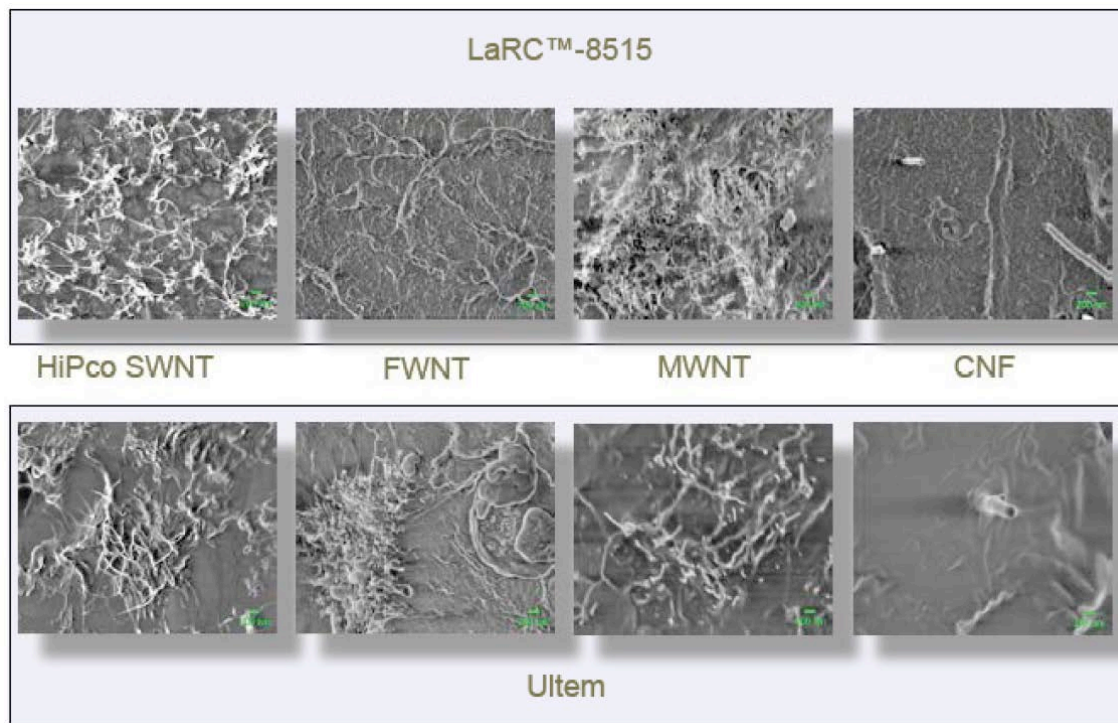


Figure 6: Effect of matrix chemistry on filler/matrix interface and dispersion.

It may also be noted, that as the nanotube diameter increases from SWCNT to MWNT, if the same nanotube length were assumed, the trend visible from the 1% LaRC™-8515 nanocomposite data is that of greater enhancement provided by the nanofiller with the larger aspect ratio.

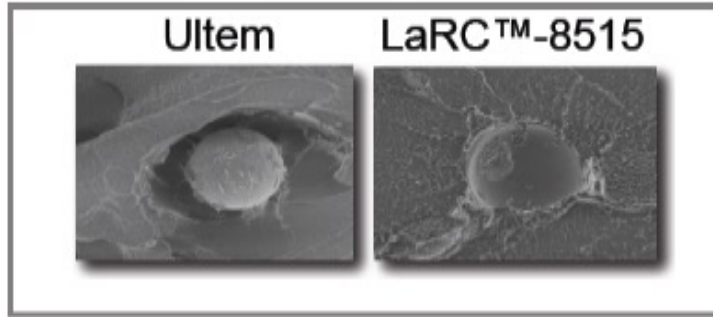


Figure 7: Comparison of Carbosphere/matrix interface for Ultem[®] and LaRC[™]-8515 nanocomposites.

Electrical Properties of Nanocomposites

The effect of the nanofillers on the electrical conductivity of the nanocomposites is summarized in Figure 8 which plots the change in conductivity as a function of nanofiller used. The % change was calculated from the difference between the conductivity of the nanocomposite and the unfilled neat matrix resin. The data show that the most effective nanofillers for electrical

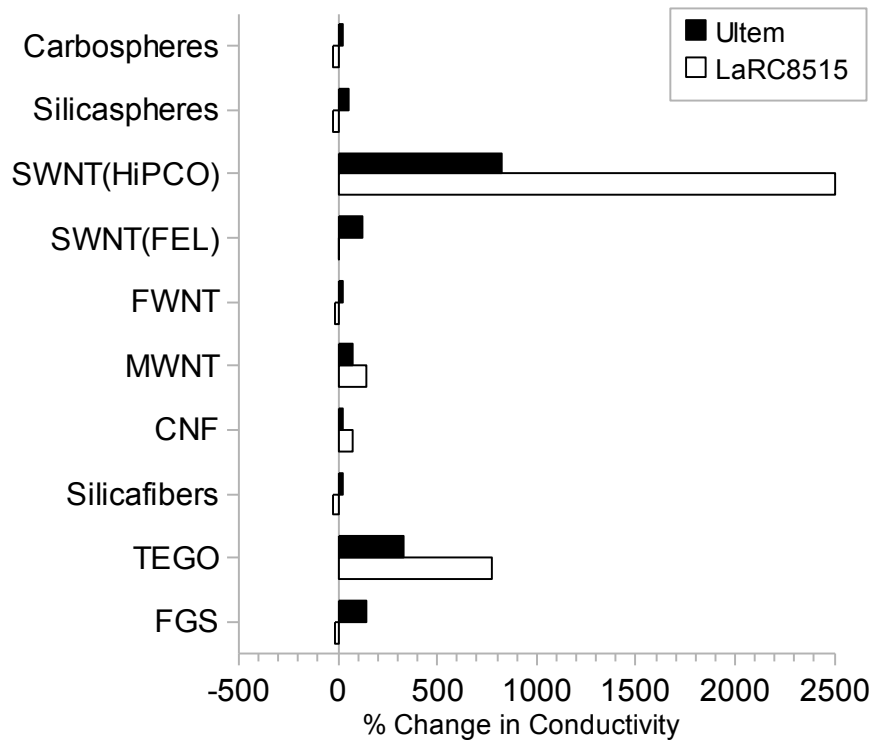


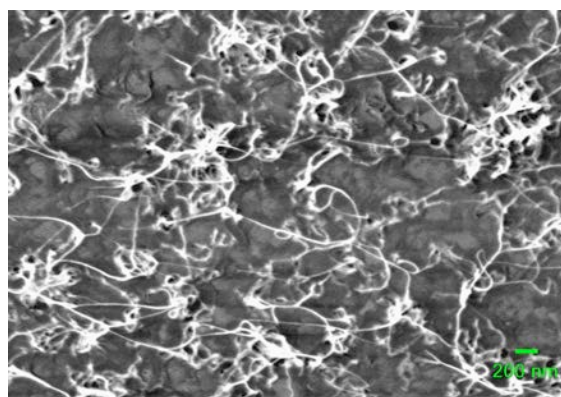
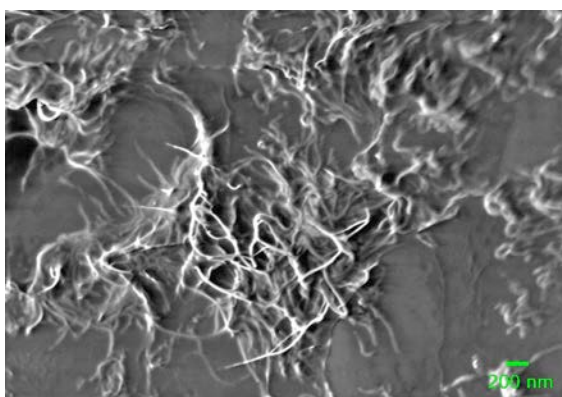
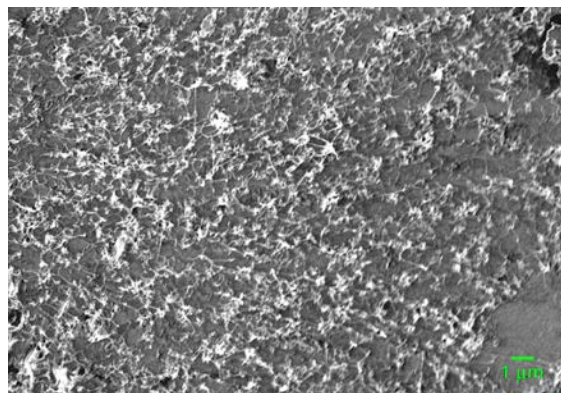
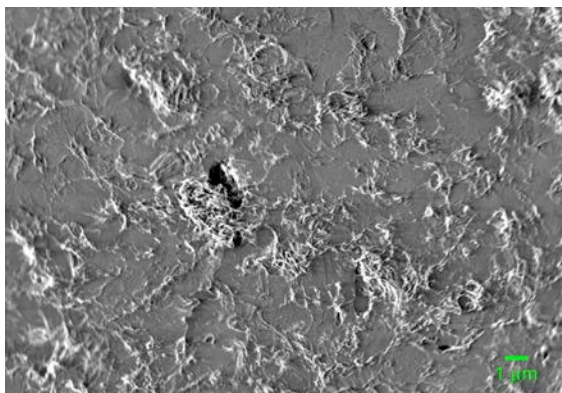
Figure 8: Change in conductivity at 1000 Hz for 1% Ultem[®] and LaRC[™]-8515 nanocomposites.

conductivity enhancements are HiPco SWCNTs and TEGO. The conductivity enhancement is most pronounced in the HiPco filled LaRC™-8515 nanocomposite and its effectiveness is confirmed by the micrographs shown in Figure 9. The images show that there are larger agglomerates in the Ultem® nanocomposites compared to the LaRC™-8515 nanocomposites. Moreover, the bottom right image for LaRC™-8515 was taken using polytransparent imaging. This allows imaging of the nanocomposite under high acceleration voltage conditions to enable a view of the CNT dispersion through the thickness of the sample, because the matrix becomes “transparent.” This type of imaging is only possible when the samples are sufficiently conductive to allow application of the high voltage without destroying the sample. Although the data in Figure 8 show that 1% SWCNT resulted in an enhancement of conductivity in the Ultem® nanocomposite, the increase in conductivity was not sufficient to permit polytransparent imaging. All samples were found to be below the conduction percolation threshold.

The effect of nanofiller doping on dielectric permittivity of the nanocomposites is summarized in Figure 10. As expected, the carbon-based fillers are more effective at increasing the dielectric permittivity compared to the silica fillers. Furthermore, within the carbon nanofiller family, HiPco SWCNTs were the most effective fillers for both Ultem® and LaRC™-8515 nanocomposites, followed by TEGO filled LaRC™-8515. In these two cases, the LaRC™-8515 nanocomposites had higher permittivities compared to the Ultem® nanocomposites. This may be attributed to the better filler/matrix interface in LaRC™-8515 nanocomposites as discussed previously. It appears that MWNTs are also effective at improving dielectric permittivity and the enhancements may be improved if processing conditions were optimized. This is a less expensive alternative to SWCNT fillers. Also interesting is that, aside from SWCNT, the 2-dimensional TEGO sheets were more effective at enhancing dielectric permittivity than 1-dimensional fillers, suggesting that more work to investigate the potential of these nanofillers is warranted.

Ultem[®]

LaRC 8515



Polytransparent imaging

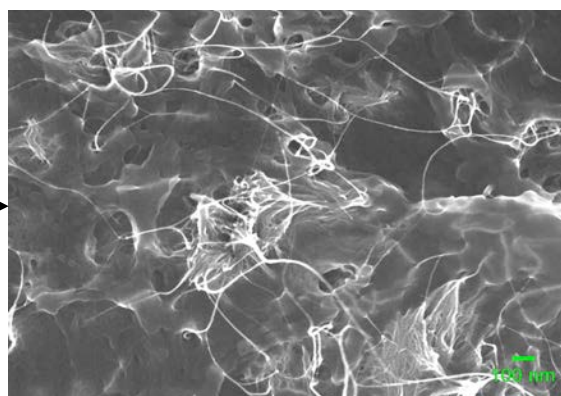


Figure 9: Comparison of conductivity enhancement by SWCNTs in Ultem[®] and LaRC[™]-8515 nanocomposites.

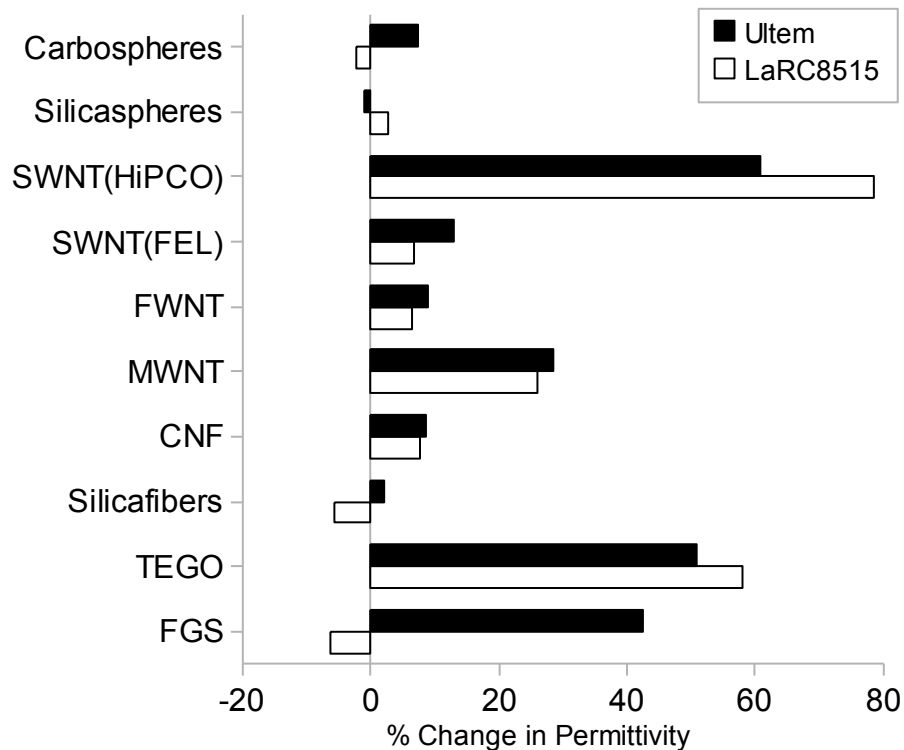


Figure 10: Changes in dielectric permittivity as a function of 1% nanofiller doping for Ultem[®] and LaRCTM-8515 nanocomposites.

SUMMARY AND CONCLUSIONS

This survey of the effect of nanofiller characteristics on nanocomposite properties demonstrated the importance of matrix composition, nanofiller quality and processing conditions on the resulting properties of the nanocomposites. The best combination of properties was obtained for the HiPco SWCNT filled LaRCTM-8515 nanocomposites. Based on an examination of the filler/matrix interface of these materials via microscopy, it can be concluded that the chemistry of the matrix is crucial to determining the quality of the filler/matrix interface, and subsequently, the mechanical and electrical properties of the resultant nanocomposites. The quality of nanofillers is very important as well. The best nanofiller in the set studied was purified HiPco SWNT. Although these nanotubes were very similar to FEL-SWNT, the properties of the nanocomposites from the purified material were superior, thus reinforcing the notion that it is critical to have consistently high quality nanofillers. Finally, optimized processing conditions have a significant impact on the quality of nanocomposites produced. Further work will be

needed to optimize the processing conditions, as well as to elucidate the mechanism for property enhancements at the molecular level.

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